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Some Features of Using the Method of Surface Plasmon Resonance for Analysis of Water and Other Liquid Substances

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Abstract
Adduced in this review are the parameters of liquid substances that influence on the main metrological characteristics of devices based on the surface plasmon resonance (SPR) phenomenon. It has been shown that the essential features of this method, when analyzing water and other liquid substances, are the dependence of sensor sensitivity and width of the sensitive area in the subsurface region of sensitive element on the refraction index of studied substances and the wavelength of laser radiation. These features cause the necessity to provide thermal stabilization of facility, and when the liquid has high viscosity and refraction index, one should dilute it. For qualitative analysis of water and other liquid media, it is recommended to apply numeric methods based on the Fresnel equations and Jones scattering matrixes.

Keywords: surface plasmon resonance; water; liquids; analysis

Introduction
Further development of such fields as pharmacology, medicine, food industry and monitoring of the ambient medium needs detection of chemical and biological substances in various media. Solution of these tasks is based on creation of new reliable analytical devices and development of the methods for detection of chemical compounds and biological objects in various liquids.
One of the promising directions in designing these analytical devices is application of surface electromagnetic waves in nanostructures, which is related with appearance of resonance phenomena, for instance SPR. Sensoric equipment based on this phenomenon possess high sensitivity to low concentrations of studied substances, which enables to use them as precision analytical devices for lab investigations in food, chemical, pharmaceutical industries, in agriculture, medicine, ecology [1]. Contrary to the refractometric method based on determination of the refraction index inside the bulk of studied substance, the SPR method enables to determine this index only in a subsurface layer over the metal film, i.e., within the range of decaying field inherent to surface plasmon. Metrological characteristics of SPR devices are subjected to the influence of many factors, namely: geometry of the optical setup, degree of polarization of laser radiation and its wavelength, proportion between indexes of refraction inherent to a dielectric substrate and studied liquid, conditions for measurements (temperature, humidity, pressure, viscosity and volatility of studied substance), processes of sorption/desorption on the surface of sensitive element. All these factors should be taken into account when performing metrological testing the liquid media by using the SPR method.
Adduced in this review are the parameters of liquid substances with different physical properties that influence on the main metrological characteristics of analytical devices based on the SPR phenomenon as well as ways to reduce their influence.

Excitation and observation of surface plasmon resonance
SPR arises in a thin metal film with the negative dielectric permittivity and high electric conduction, when this film, being a sensitive element, covers a transparent dielectric

substrate. This phenomenon is observed when the angle of light incidence onto this film corresponds to total internal reflection at the boundary "film – substrate". Under these conditions, monochromatic laser radiation excites conduction electrons in the metal film. It causes oscillations in the electron gas density in metal and appearance of sign-alternating charges in its subsurface layer. The changes in the surface potential induces surface electromagnetic wave (surface plasmon) that propagates along the boundary "metal – studied substance". Excitation of surface plasmons is observed as a sharp drop in the intensity of reflected light for a definite angle of laser radiation incidence. This angle is called resonant. From the analytical viewpoint, it is important that the value of this resonant angle depends on the refraction index and concentration of substance on the surface of sensitive element that contact with the studied liquid. As a metal sensitive layer, gold is preferably used as metal with high conduction and chemical inertness.

The most widely used method for excitation of surface plasmons is realized via a coupling prism. In this case, to provide total internal reflection, the following condition should be satisfied: the refraction index of studied substance has to be lower than that of the coupling prism, i.e., $n_d < n_p$. There are two optical setups to realize this method of excitation, namely: Kretschmann geometry [2] and Otto one [3]. In the Kretschmann geometry, under the condition of total internal reflection, the prism with a high refraction index n_p is coupled with a metal-dielectric waveguide that consists of a film possessing the dielectric permittivity ε_m and thickness d as well as semi-infinite dielectric with the refraction index n_d, the role of which is played by the studied substance (Fig. 1a). This scheme is more often used, because it is easier-to-produce in practice.

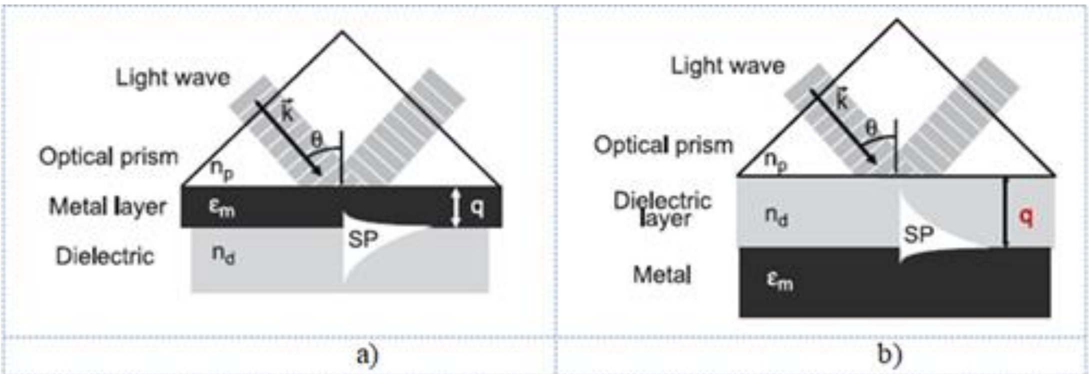
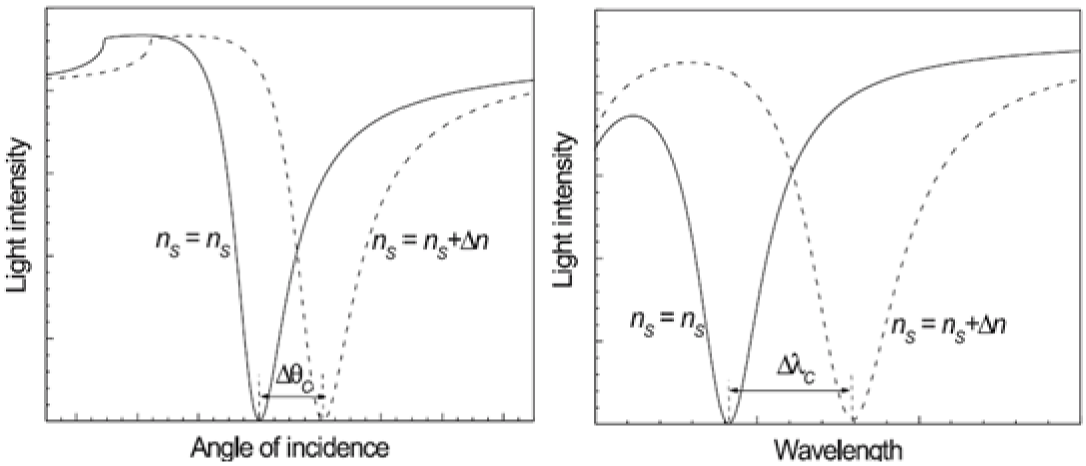


Fig. 1: Scheme of excitation of surface Plasmon's in the Kretschmann geometry (a) and Otto one (b) under condition of total internal reflection [4].

The change in the refraction index of the studied medium causes the respective change in the frequency of surface plasmons, which results in changes of resonance conditions: the resonant angle or resonant wavelength is changed. In the devices with angular scanning, the monochromatic electromagnetic wave excites surface plasmons within the definite range of angles of radiation incidence on the boundary metal – studied medium. The incident wave transfers the energy of its photons to electrons in the metal film. In this case, one can observe a minimum in the reflected light intensity (Fig. 2a) [4]. In SPR facilities with spectral scanning, surface plasmons are excited by light containing various wavelengths. The angle of their incidence onto the metal film is kept constant. Coupling between the incident light wave and surface plasmons can be observed for various wavelengths, but there is only a narrow interval of wavelengths when one can observe the most efficient transfer of the wave energy to the plasmon sub-system, the minimum intensity of reflected light being observed at the moment. This minimum serves as the sensor output signal (Fig. 2b) [4].



a) b)

Fig. 2: Angular (a) and spectral (b) dependences of the reflected light intensity at the boundary sensitive element – studied substance before (—) and after (---) changing the refraction index of the substance by the value Δn [4].

Excitation of SPR becomes especially efficient under the following conditions:

1. light incident onto the metal surface is plane-polarized;
2. electric vector of electromagnetic wave belongs to the plane of incidence, while the magnetic vector lies in parallel to the metal surface;
3. Projection of the light wave vector on the metal film plane is equal to the surface plasmon wave vector.

Main metrological characteristics of facilities based on SPR

This section should present your findings objectively, explaining them largely, concisely, precisely in the text. You should show how your results contribute to the scientific knowledge inacademic communityclearly and logically.Results and Discussion may be divided by subheadings, concluding equations, figures, tables, etc.

Sensitivity

The sensitivity of devices designed in accord with the Kretschmann geometry with the fixed light wavelength is defined as the ratio of resonance angle shift Δθmin to the change in the refraction index of the studied substance ΔNa (1) that causes this angular shift [5].

$$S_\theta = \frac{\Delta\theta_{\min}}{\Delta N_a} \quad (1)$$

where, S_θ is the device sensitivity, when measuring the shift of resonance angle;

$\Delta\theta_{\min}$ – value of the resonance angle shift, degrees;

ΔN_a – change in the refraction index of the studied substance.

The change in the resonant angle $\Delta\theta_{\min}$ is proportional to the concentration ΔC of the studied substance inside the analyzed volume. Therefore, in solutions the SPR sensor reacts only on changes in the refraction index and absorption of medium. In colloidal solutions, one can observe adsorption of solution components on the sensor surface, which results in increasing its concentration in the sensitivity range as compared to that in the bulk. Therefore, the sensor reacts both on changes in refraction (absorption) indexes and the thickness of adsorbed layer on the sensitive element surface. The total change in the refraction index Δn_b [4] of the studied substance (2), when its molecules are adsorbed on the sensitive element surface, depends on the augmentation of the volume refraction index (d_n/d_c)vol and can be expressed through the solution concentration [4].

$$\Delta n_b = \left(\frac{dn}{dc}\right)_{vol} \Delta c_b , \quad (2)$$

where, Δc_b is the concentration of the bound analyte in milligrams per milliliter[4].

The value of volume refraction index depends on the structure of analyte molecules and varies within the range 0.1 to 0.3 ml/g (3). If adsorption of the studied substance takes place in a thin layer of the thickness h on the sensitive element surface, then the total change in the refraction index can be expressed via the refraction index and surface concentration (3).

$$\Delta n_b = \left(\frac{dn}{dc}\right)_{vol} \frac{\Delta\Gamma}{h} , \quad (3)$$

where, ΔΓ is the surface concentration in mg/mm^2 [4].

The degree of adsorption depends on the sensitive element material and properties of its surface (roughness, porosity and so on). The increase in porosity of the surface enables to enhance the sensor response [6]. The sensitivity of SPR method also depends on the width and shape of the resonance characteristic, which defines the accuracy in registration of its minimum position. Thereof, the possible way to considerably enhance the SPR sensor sensitivity consists in applying the techniques of narrowing its resonance characteristic. It can be reached by placing diffraction or textured structures inside the range of plasmon decay on the surface of sensitive element [7] as well as by application of bi-metal layers silver-gold [8] and increasing the wavelength of laser radiation exciting SPR [9].

Limit of detection

The resolution capability is defined by the detection limit, i.e., minimum change in the analyte refraction index ΔNa_{MIN} (or in the respective concentration) that can be determined using this SPR device. It is also known that the detection limit is defined by the device sensitivity value and the noise level χ in its information parameter (4) that is the change in its minimum angle $\Delta\theta_{\min}$.

$$\Delta N_{aMIN} = \frac{\chi_\theta}{S_\theta} \quad (4)$$

where, ΔN_{aMIN} – the device detection limit;

χ – noise level in the base line of measured signal, degrees.

Ligh passing through the elements of the device optical system increases the noise component as a result of varying optical and geometrical parameters of these elements. They are the source for excitation of surface plasmons in thin metal film and photodetectors for measuring the intensity of reflected light.

As a light source, they usually use a semiconductor laser diode, and parameters defining the noise level are oscillations in its radiation intensity and wavelength, which depend on the value of current passing through the emitting crystal of this diode as well as on the geometrical dimensions of its optical resonator [10]. Like to each semiconductor, the current value for the constant supply voltage is defined by the crystal temperature, the increase in which enhances the density of minor charge carriers and causes the growth of total current. The increase in temperature has an effect on resonator geometrical parameters due to its volume expansion, which causes the changes in the operation wavelength and, with the following increase, results in failure of oscillations – the laser loses its coherency.

In most of cases, a semiconductor silicon photodiode serves as a photodetector. The noise of this photodetector is related with random fluctuations in its conduction and has three components defined by physical mechanisms of these fluctuations: thermal noise, shot noise and the generation-recombination one [11]. The level of thermal noise is in proportion to the semiconductor temperature. The shot

noise is related with the current value in semiconductor. The generation-recombination noise is proportional to the rate of creation of minor charge carriers in semiconductor, which increases with the temperature growth.
Convection fluxes of the studied substance in the measuring cell of SPR device and oscillations of microscopic bubbles on the surface of metal film in the sensitive element can be considered as additional sources of noises.
As the temperature changes are the main reason for noise, in the majority of SPR devices designers foreseen constructive and program means to reduce its influence. To these means, one can relate complex and local thermal stabilization [12] and thermal compensation [13]. The latter can be realized using the numeric methods for processing the measurement results concerning the changes in the refraction index of the studied substance.

Accuracy of measurements

The accuracy of measurements is defined by the value of errors in the obtained results. In relation with the source of their origin, one can separate three groups of reasons and, respectively, three complex components in the measurement error: methodical, instrumental and readout ones [14]. The methodical error is caused by deficiencies of the measuring method and ways for using the measuring means. In the case of indirect measurements, the methodical errors are caused by regularities that relate the measured values and constant parameters with the final result of measurements, which is obtained by an indirect way. The instrumental error is caused by deviation in optical and geometrical parameters in the device construction elements. To the components of instrumental error, one should relate the error in positions of mechanism responsible for angular scanning or the wavelength one (rotation of the prism or monochromator). This error can grow in the process of SPR facility exploitation as a result of parts wear in the rotating mechanism and thermal expansion of elements in its construction. The readout error consists of two components: i) the error of analog-digit transformation performed over the measured values of intensity of light reflected from the sensor, and ii) the error in determination of the angular position inherent to the minimum of reflection characteristic. The latter error can be reduced by mathematical processing the measurement results [15, 16].
Thus, there are several principal constructive-and-technological factors that influence on the nature and value of the main errors in measurement results, namely: the sensor construction and technology of its production, temperature mode of its operation, excitation wavelength, rate of pumping the studied substance through the measuring cell, parameters of kinematic scheme, and method of mathematical processing the results of measurements [17].

Parameters of water and liquid media influencing the metrological characteristics of facilities based on SPR

The main parameters of liquid media, which influence on metrological characteristics of SPR devices, are as follows: the refraction index, its temperature coefficient and dispersion, viscosity of the studied sample, and wettability of the sensitive element surface by it.
The value of refraction index inherent to the studied substance Na influences on the sensitivity of SPR devices, since the magnitude of it depends on the ratio of Na and Nп values (5) [5].

$$S_\theta \equiv \frac{d\theta}{dn_a} = \frac{\varepsilon_{mr} \cdot \sqrt{-\varepsilon_{mr}}}{(\varepsilon_{mr} + n_a^2) \cdot \sqrt{\varepsilon_{mr}(n_a^2 - n_p^2) - n_a^2 \cdot n_p^2}}, \quad (5)$$

Where, S_θ – the device sensitivity in the mode of measuring the resonance angle shift;
$\Delta\theta_{min}$ – value of the resonance angle shift, degrees;
N_a – refraction index of the studied substance;
N_p – prism refraction index;
ε_{Mr} – real component of dielectric permittivity of metal in the sensitive element.

The increase in N_a value results in reducing the sensitivity, which introduces additional non-linearity to the measured concentration characteristics of the studied substances. Besides, the value of dielectric permittivity inherent to the studied medium defines the depth of penetration of the surface plasmon field into the medium δ_d and metal film δ_m (6).

$$\delta_d = \frac{\lambda}{2\pi} \cdot \sqrt{\frac{\varepsilon_a + \varepsilon_{mr}}{-\varepsilon_a^2}} \cdot \quad \delta_m = \frac{\lambda}{2\pi} \cdot \sqrt{\frac{\varepsilon_a + \varepsilon_{mr}}{-\varepsilon_{mr}^2}}; \quad (6)$$

where, δ_d is the depth of plasmon field penetration into the studied medium, m;
λ – wavelength of laser radiation, m;
ε_a – dielectric permittivity of the studied medium;
ε_{Mr} – real component of the dielectric permittivity of metal in the sensitive element.

Thus, the value N_a defines the range of sensitivity for this method and limits overall dimensions of additional functional layers and/or receptors for biomolecular analysis on the surface of sensitive element metal film. In the case when these dimensions exceed the value δ_d, the method becomes non-sensitive to changes in the refraction index of the studied substance. Therefore, when analyzing liquid media with a high refraction index (> 1.4), to reduce its influence on the sensitivity it is recommended to use dilution in a set proportion [18].
The temperature coefficient of refraction index defines the degree of influence of temperature changes in the studied substance on the respective changes in the refraction index. The dependence of refraction index on temperature causes the temperature drift of the base line, when measuring kinetics of chemical and biological processes on the surface of SPR sensor sensitive element, which introduces essential errors into measurement results and complicates their interpretation. Estimation of the temperature influence was performed in [19], where it was shown that the contribution of temperature factor into the total absolute error in measurements of the refraction index can reach more than 50%. For example, in the case of water that is widely used as the main solvent for chemical and biological reactions, the temperature coefficient of refraction index is close to $-1 \cdot 10^{-4}$ K^{-1}. In the case of gases, this value is considerably lower $(0.2 \ldots 1) \cdot 10^{-5}$ K^{-1}. Therefore, when studying water and other liquid media by using the SPR method, it is

especially important to take the influence of this parameter into account. To reduce the influence of temperature factor, they use local stabilization of the studied substance temperature. With this aim, one can place a regulated heater or cooler directly in the measuring cell of SPR device, which contains the studied substance. The most efficient way to realize temperature stabilization is application of film heaters located directly under the metal film of sensitive element as well as using the system for complex cooling the device as a whole, which provides the maximum speed of response and efficiency of thermal stabilization [20].

Dispersion of the refraction index has an effect on the accuracy of measurements and detection limit [5]. Deviation of the laser radiation wavelength λ is related with changes in the refraction index of the studied substance. Gradual changing the value λ (with the time constant higher than 1 min) causes the dispersion drift of the base line when measuring the kinetic of surface processes, while the fast changing λ value (with the time constant lower than 10 ms) enhances the noise of base line, which lowers the resolution capability of SPR device. The reasons for changing the radiation wavelength are both the temperature factor and poor stabilized laser power supply. To reduce the influence of refraction index dispersion, it is reasonable to use local stabilization of laser and enhance stability of its power supply.

The viscosity of substance and the studied liquid wettability of the sensor element surface influence on the sensitivity and accuracy. This influence is clearly pronounced when using the sensitive elements with an additional external porous layer that is usually used to enhance the sensitivity. Viscous liquid cannot penetrate into pores, and the latter remains filled with buffer, the refraction index of which differs from that of the studied liquid. It causes lower response of the sensor as compared with case of full contact of liquid and the surface of sensitive element metal. To lower the viscosity, like to reducing the refraction index of liquid, it is recommended to use dilution. The influence of low wettability of the sensitive element surface by the liquid is similar to the effect of viscosity. When applying water and its solutions to enhance surface wetting ability, they use additional polymer coatings formed by polymerization in the high-frequency plasma of inert gas [21].

Features of analyzing water and liquid media by using the SPR method

To quantitatively analyze water and liquid media, when qualitative parameters are known (for example, the nature of impurities), one can use mathematical methods for this analysis. Water or other liquids suspension with solid nano-sized particles (filler or impurities) can be considered as the model heterosructure consisting of a liquid matrix and solid-phase dispersed filler. Knowing the values of the relative dielectric permittivity for these matrix and filler, one can determine the heterostructure percentage. The most widely spread approaches to describe this system are the Maxwell-Garnett and Bruggeman ones that are valid for media with chaotical distribution of filler in matrix. The model based on the Bruggeman approach is applicable in the case when the volume fractions of components f_1 and f_2 correspond to the ratios from 1/3 up to 2/3, i.e., for high concentrations. Since in accord with the set task it is necessary to control low concentrations of nanoparticles in water and liquid suspension by using the SPR method, the best choice for modeling is the Maxwell-Garnett approach.

Adduced in the work [23] are the results of numeric modelling and experimental confirmation of the SPR-sensor response dependence on low concentrations of water suspensions prepared from nanoparticles of diamonds, silicon dioxide, iron and its oxide that differ by their optical properties. It has been demonstrated the efficiency of using the Maxwell-Garnett approach as well as formalism of the Jones scattering matrixes in calculations of the SPR-sensor response. In paper [24] shown the possibility to diagnose quality of motor oil by using the SPR-method for comparison of optical refraction indexes inherent to the initial fresh oil and to that used for different exploitation terms. This comparison and Maxwell-Garnett approach enabled authors to determine not only the motor oil degradation level but also availability of wear particles from motor parts. Investigated in this work were four samples of the motor oil Genuine 5w-30 dexos 2, namely: the fresh one and three ones taken after car mileages 180, 430 and 712 km. Motor oil is high refractive index liquid: is 1.45…1.50. By authors has been experimentally shown that using the SPR method improves more than one order (from Δf_{min} = 0.17 % vol. down to Δf_{min} = 0.0107 % vol.) the detection limit and enhances sensitivity of measuring the wear particles concentration in motor oil as compared with the known refract metric method. To increase the sensitivity, the authors applied dilution of engine oil samples with a solvent. Application of the optimum concentration (50%) for the solution of this oil in rafinate of benzene reforming provides the 3-fold increase in the sensitivity and selectivity. Thus, it is experimentally proved that the SPR method can be offered to control quality of motor oils as well as the degree of wear inherent to interacting parts of machinery.

Thus, the application of the surface Plasmon resonance method in conjunction with numerical methods, such as the Maxwell-Garnett approach, allows one to analyze liquids with different refractive indexes from 1.33 (water) to 1.5 (motor oils), determining the presence of impurities in them. Moreover, the concentration of impurities is an order of magnitude lower than for the refract metric method on the basis of total internal reflection. To reduce influence on the sensitivity in case analyzing liquid media with a high refraction index (> 1.4) it is recommended to use dilution in a set proportion.

Conclusions

The main parameters of liquid media influencing the metrological characteristics of SPR devices are as follows: refraction index, temperature coefficient of the latter and its dispersion, viscosity of the studied liquid, and wettability of sensitive element surface by this liquid. The increase in the refraction index value results in lowering the sensitivity, which introduces additional non-linearity into the measured concentration characteristics of the studied substances. Therefore, when studying liquid media with a high refraction index, it is recommended to use dilution in a set proportion. The temperature coefficient of refraction index defines the degree of influence of temperature changes in the studied substance on the respective changes in its refraction index. Concerning water that is used as the main solvent for chemical and biological reactions, the temperature coefficient of refraction index is close to – $1 \cdot 10^{-4}$ K^{-1}, therefore, when studying water and other liquids by using the SPR method, it is especially important to take the influence of this parameter into account. To reduce the effect of this factor, it is offered to apply thermal stabilization of the studied liquid. Deviation of the laser radiation wavelength exciting surface plasmons is related with a change in the refraction index of the studied substance. The reasons for changing the radiation wavelength are both temperature factor and poor stabilized laser power supply. To reduce the influence of refraction index dispersion, one can use local stabilization of laser temperature as well as stabilization of the power supply voltage. To control low concentrations of nanoparticles in water and liquid suspensions by using the SPR method, the best choice for modeling is the Maxwell-Garnett approach. The application of the surface plasmon resonance method in conjunction with numerical methods, such as the Maxwell-Garnett approach, allows one to analyze liquids with different refractive indexes from 1.33 (water) to 1.5 (motor oils), determining the presence of impurities in them. Moreover, the concentration of impurities is an order of magnitude lower than for the refractometric method on the basis of total internal reflection. To reduce influence on the sensitivity in case analyzing liquid media with a high refraction index (> 1.4) it is recommended to use dilution in a set proportion.

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